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MICRO-COMPRESSION TESTING OF FCC METALS : A SELECTED OVERVIEW OF EXPERIMENTS AND SIMULATIONS (PREPRINT)

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Micro-compression tests allow for the direct measurement of stress-strain behavior in volumes of material that have micro-scale dimensions. Initial studies worldwide have focused on the exploration of size-scale effects, where sample dimensions at the micrometer- and sub-micrometer scale can dramatically affect the fundamental processes of plastic deformation. Importantly, this scale of test volume can be directly modeled using state-of-the-art discrete dislocation simulations, the results of which have been essential to understanding the changes that can occur to dislocation mechanisms within small volumes. This combination of miniaturized testing and modeling that closely mimics these experiments provides a new pathway to characterize plastic flow on a highly localized basis.

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Micro-compression testing of FCC metals: a selected overview of experiments and simulations

Michael D. Uchic, Paul A. Shade, Dennis M. Dimiduk

Summary: Micro-compression tests allow for the direct measurement of stress-strain behavior in volumes of material that have micro-scale dimensions. Initial studies worldwide have focused on the exploration of size-scale effects, where sample dimensions at the micrometer- and sub-micrometer scale can dramatically affect the fundamental processes of plastic deformation. Importantly, this scale of test volume can be directly modeled using state-of-the-art discrete dislocation simulations, the results of which have been essential to understanding the changes that can occur to dislocation mechanisms within small volumes. This combination of miniaturized testing and modeling that closely mimics these experiments provides a new pathway to characterize plastic flow on a highly localized basis.

Introduction: Uniaxial mechanical tests are perhaps the most commonly performed deformation experiments that provide basic design information. The popularity of these tests can be traced to the relative uniformity of the stress-state within the active region of the sample, which greatly aids in the subsequent interpretation of the test data. Tension tests are generally preferred because the stress-state in the gage section is more uniform compared to compression experiments, although compression experiments have selected advantages that include a simple and efficient sample shape, and a relatively uncomplicated procedure to place the sample within the loading train.

Shrinking uniaxial tension and compression test methods to the micro-scale provides a unique opportunity to study plastic deformation. One potential benefit is that the dimensions of the sample volume can be of similar size to the fundamental length scales for dislocation-based plastic flow. One can imagine that the processes of dislocation multiplication, annihilation, storage, glide, percolation, and so on, each has a characteristic length associated with that particular phenomenon.[1] When the sample dimensions approach these length scales, the proximity of the free surfaces potentially affect or alter these processes, which can result in observable changes in the material flow behavior. Another potential advantage is that the finite sample volume can enable studies that probe the effect of local heterogeneities. For example, one can extract small samples from site-specific locations within a bulk crystal, where these samples might contain local changes in either chemistry or variations in microstructure (i.e., dislocation, grain, precipitate, void, defect, or other features), or both. Through testing of these isolated samples, one can more readily discern the influence that these internal variations have on mechanical properties, or help identify 'weak-links' within the microstructure. In addition, another advantage of these micro-scale experiments is that they are amenable to study using sophisticated modeling methods such as 3D discrete dislocation simulations (DDS), where the entire test volume can be examined with ever-increasing fidelity. The 3D DDS can calculate the force-activated motion of all dislocations within the diminutive sample, allowing one to both visualize and quantify the dislocation activity that is responsible for plastic flow.

There are excellent examples of small-scale tension test methodologies in the literature that produce a well-understood and uniform stress-state in samples with micro-scale dimensions.[2-5] However, these

methods typically require samples produced by microelectronic processes, e.g., freestanding thin film samples that remain attached to a substrate. While this eases the difficulty in preparing small test volumes, these samples and corresponding microstructures are limited to materials and processing conditions available in microelectronic fabrication facilities. In order to circumvent this restriction, some of the present authors (along with Jeff Florando and William Nix) developed a micro-compression test methodology about six years ago.[6-9] In its present form, the methodology uses micro-machining methods to produce compression samples within the surface of bulk materials, which are subsequently tested using a flat punch. The technique potentially allows one to explore deformation at small scales in a wide range of materials that can have complex chemistries, internal microstructures, and defect structures.

In this article we will highlight selected research that has utilized micro-compression testing or discrete dislocation modeling to examine the small-scale behavior of pure FCC metals. We draw upon work that we have been personally associated with, as well as other published studies.

Micro-compression Test Method

The micro-compression test methodology scales the conventional uniaxial compression experiment to the micro-scale using commercially-available laboratory equipment.[6-9] A schematic of the test geometry is shown in Figure 1a, which highlights the relationship between the sample and a nanoindentation system that typically acts as the mechanical test frame. Other than the size-scale, the only significant difference between the conventional compression test and the micro-compression test is that there is no lower compression platen underneath the active gage section. Instead, the sample is integrally-attached to the bulk substrate, and the transition region between the sample-and-substrate effectively acts as the lower platen. This sample geometry eliminates the need for micromanipulation equipment to place the sample within the test frame, which helps tremendously in the practical execution of the test. Also, the load and displacement ratings and resolution of many commercial nanoindentation systems are ideal for performing micro-compression experiments.

Micro-compression samples have been typically fabricated by focused ion beam (FIB) micromilling of bulk samples, [6-9] but alternative methods using larger-scale machining methods,[8] micro-electronic based[10] or other growth methods[11] have also been advanced to overcome limitations with FIB-based processing. Sample sizes have ranged from 0.25 to 80 micrometers in diameter, with corresponding gage lengths that varied from 1 to 160 micrometers. One significant advantage of FIB-prepared samples are that these can be fabricated at very precise locations on a sample surface, such as within a single grain of a polycrystalline alloy, which potentially allows for measurement of 'single crystal' properties from polycrystalline materials. Disadvantages for FIB-prepared samples can include lengthy milling times and potential effects from the thin irradiation zone at the outer sample surface that remain a subject of current study.[10-15] An example of a FIB-micromachined and tested sample is shown in Figure 1c and 1d.[9]

Once fabricated, the samples are loaded into the nanoindentation frame, an individual sample is identified for testing, and the flat-punch tip is brought into contact with the top surface of the

microsample in order to compress the sample volume. Prior to testing, the dimensions of the sample cross-sectional area and gage length are obtained, as these are needed to convert the load-displacement data into a stress-strain curve using the standard formulae for compression tests. These dimensions are usually determined from Scanning Electron Microscope images. Examples of both load-controlled and displacement-controlled testing can be found in the literature, although most experiments have focused on performing constant displacement rate tests where the initial strain rate is approximately 10^{-3} to 10^{-4} s^{-1}.

Experimental Results for FCC Metals

Figure 2 contains stress-strain data from representative micro-compression experiments conducted at room temperature on single-slip oriented pure Ni microcrystals that contain a moderate starting dislocation density (ρ_o ~ $5x10^{12}$ to $1x10^{13}$ m^{-2}).[7,9,16] From this figure, one can observe that the mechanical response of FCC microcrystals differ from bulk single crystal behavior in the following ways. First, substantial strengthening is observed in microcrystals with decreasing sample volume, where this strengthening typically occurs at small-to-moderate plastic strains (< 5-10%). Second, the plastic flow response is intermittent, and is composed of regions of easy glide with little-to-no strain hardening that are separated by regions of rapid hardening at elastic or nearly-elastic rates. Third, the flow curves vary stochastically, and the variation in flow behavior intensifies with decreasing sample volume. All of these features are typical hallmarks of microcrystal flow. In the following section, we address these phenomena.

Size-dependent strengthening

A closer inspection of Figure 2 shows that the increase in flow stress for microcrystals results from both an increase in the proportional limit, and enormous strain hardening rates at micro to moderate strains.[7,9,16] These enhanced strain-hardening rates are often greater than Stage II hardening (which is normally the highest strain hardening rate observed for bulk experiments on FCC crystals), and can approach a significant fraction of the elastic modulus.[9,16] This 'enhanced' strain hardening behavior is size-scale dependent, with smaller samples exhibiting higher strain-hardening rates.[9,16,17] Taken to the extreme, pure-metal microcrystals with nanoscale dimensions can exhibit tremendous size-dependent strengthening. For example, the studies by Frick et al.[17] and Shan et al.[15] report that 200 nanometer diameter, multiple-slip oriented pure Ni microcrystals can support stresses of 2 GPa and higher, in contrast to bulk yield stress values for single crystal Ni that normally range from 10 MPa and higher.

One obvious question to ask is 'what is the functional form between sample diameter and flow stress?' In the regime where size-dependent strengthening is observed, the relationship between the resolved shear stress (τ) and sample diameter (d) can be empirically described by a power law, which is shown below in a form that normalizes the flow stress to provide a more accurate comparison of size-affected flow between different metals:

$$(\tau - \tau_o)\ b_{Ni} / (K_s\, b_{metal}) = B d^{-n} \qquad 1.$$

where n is the power-law exponent, B is a constant, K_s is the anisotropic shear modulus, and b_{Ni}/b_{metal} is the relative ratio of the Burgers vector of each metal to a reference (selected to be Ni for the data shown in Figure 3). Figure 3 shows the normalized flow stress versus sample diameter for all of the pure-metal FCC size-dependent strengthening data from a number of different research studies.[18] By plotting the data in this way, one can see that all of this data collapses onto a single band, which generally holds for sample diameters that range from a couple hundred nanometers to tens-of-micrometers in diameter. A scaling exponent of 0.6 is a reasonable match to much of the data, assuming the reference stress, τ_o, is negligible. Note that the scaling exponent is different than those associated with grain-size strengthening (n = 0.5) or surface-controlled nucleation (n = 1).

Size-dependent strengthening is observed in both single-slip,[7,9,19,20] and multiple-slip orientations.[10,17,21,22] Although no micro-compression study has examined the same material under both multiple- and single-slip orientations, the relative importance of crystal orientation lessens as the sample diameter shrinks to the micrometer scale, as the stress-strain curves for all orientations become qualitatively similar when size-affected behavior dominates plastic flow.

Intermittency and stochastic flow

In addition to size-dependent strengthening, another almost-universally observed feature in FCC (and BCC) microcrystals is an intermittency associated with plastic flow. By intermittency, we mean that microsamples, especially those that are single crystals, display a binary mechanical response composed of either periods of easy glide or elastic/nearly-elastic loading. Intermittent flow is usually not observed in bulk materials save for well-known cases such as the Portevin-Le Chatelier effect, but these discrete bursts are regularly observed in microcrystal tests, and are clearly apparent in Figure 2.[9] Note that the curves shown in this figure are comprised of many hundreds or even thousands of discrete events that range from Angstroms to micrometers in scale,[23] although because of the construction of this figure only the largest events are observable. Micro-compression tests offer a unique means of studying this behavior, not only because the finite sample volume allows these events to be readily identified, but also because the test allows for the displacement associated with a strain burst to be directly measured[23] (unlike acoustic methods that measure the magnitude of a strain burst indirectly). In addition, one can also determine the stress[20], time, and potentially the spatial location[24] associated with each event.

Although the strain bursts occur at stochastic intervals at stresses above the proportional limit, and the magnitude and timing of these events is also stochastic, the global statistics of strain bursts are well described by a power law. A quantitative analysis of these statistics has been performed by a number of independent studies.[20,23,25-28] These studies have correlated the frequency of occurrence of a given strain burst with its magnitude, and all agree that the number and magnitude of slip events display power-law scaling (with a cut off), in some cases spanning event sizes that range over three-orders of magnitude. This power-law scaling is described by the following equation:[26]

$$n(x) = C\, x^{-\alpha} \exp[-(x/x_0)^2] \qquad 2.$$

where $n(x)$ is the probability of an event of magnitude x, C is a constant, α is the power law scaling exponent and, and x_0 is the characteristic magnitude of the largest strain burst. The value of α is reported to be approximately 1.6 for much of the experimental data published to date, [20,23,25-28] but one study showed that this value is dependent upon the applied strain rate.[25] Some studies have sought to further quantify the relationship between slip events by examining other correlation statistics.[20] Although these types of experimental studies are relatively new, the discovery of this behavior has current practical implications on plastic forming at the micro-scale,[26] and long-term implications on the development of new meso-scale deformation theories.[23]

3D Discrete Dislocation Simulations

As mentioned in the introduction, the diminutive size of microcrystals enables the use of 3D DDS to model the entire sample volume.[26,29-36] This simulation method is well suited to study the problem of microcrystal flow, as it can naturally account for both the far-field and local interactions between dislocations, as well as accurately track the motion of dislocations, including those that are in contact with the nearby free surfaces. It is important to note that at present these methods at best mimic the microcrystal experiments, and also vary considerably in some critical areas. One particularly crucial issue pertains to the initial dislocation substructure, especially with regards to the number, size, and distribution of dislocation sources. All of the published studies contain moderate-to-high dislocation densities that are similar or greater than those found in the microcrystal experiments, and most of these studies instantiate the initial dislocation density as a set of Frank-Read sources (FRS) having rigidly-fixed ends.[26,29-35] Significantly, the strength and distribution of the initial FRS can vary significantly from study-to-study, which can strongly affect the outcome of the simulations.[18] Other differences include the following: whether cross slip is allowed, whether the influence of the free surfaces and the test boundary conditions are included, the magnitude of the applied strain rate, the loading mode, crystal orientation, sample aspect ratio, and the size of the simulation volume. In spite of these differences, the results from state-of-art 3D DDS have made a significant impact on the understanding of microcrystal deformation.

Importantly, many of these simulations display size-dependent strengthening, significant strain-hardening rates at small strains, and intermittent flow, which are all consistent with the aforementioned 'hallmarks' of microcrystal flow. [26,29-36] As an example, Figure 4 plots the size-dependent strengthening observed in the 3D DDS studies, all of which fall within the range of data that was determined from experiments.[18] With regards to the mechanisms that are responsible for this behavior, we highlight two new mechanisms[29,30] that are observed in many of the simulations.

The first new mechanism provides an increase in the strength of existing dislocation sources due to the proximity of free surfaces—a process termed *source-truncation hardening*.[29,30] Source-truncation occurs during the initial operation of a FRS, where the interaction of a single FRS source with the nearby free surfaces separates the FRS into two single-arm sources. Depending on the sample size, these single-arm sources may have minimum arm-lengths that are much smaller than the original FRS length, and thus each of these single-arm sources would require a significantly higher stress to operate than the original source. In selected simulations, spiraling single-arm sources have been observed to account for almost

all of the plastic strain,[29,34-36] and thus the flow stress is controlled by the largest single-arm sources and their respective Schmid factors.[29] A statistical analysis of the source-truncation process has shown that this process can account for much of the size dependent strengthening that is observed experimentally.[37]

The other new size-dependent mechanism identified by three independent studies[29,33,36] is related to the paucity of dislocation sources in microcrystals. Unlike bulk crystals that have an almost infinite supply of potential sources, microcrystals have only a finite number of available sources, and one can imagine that the plastic flow of microcrystals is therefore more sensitive to the termination of an active source due to typical forest-hardening processes. If this happens, the stress increment required to activate the next-weakest source may be much larger than would be required in a bulk crystal, simply because of the limited number of possible sources that the sample has to choose from. The reduced number of 'weak-links' in the dislocation microstructure can result in much more potent average strain-hardening rates, especially in the small-strain regime. Consequently, the flow behavior in many FCC microcrystals takes place intermittently via the sequential activation and obstruction of the weakest sources, and this process is termed *exhaustion hardening*.[9,29] By this mechanism, a micrometer-scale crystal may be 'starved' of mobile dislocation density, even at sizes where the effects of images forces and free surfaces are dramatically reduced. This mechanism of enhanced forest hardening is believed to be responsible for the strengthening effects extending to microcrystal sizes exceeding 20 micrometers in diameter.[29]

The combination of source-truncation and exhaustion hardening convincingly accounts for the observation of stochastic flow in 3D DDS. For the same starting density, different instantiations of the initial dislocation substructure result in changes to both the distribution of largest single-arm source lengths, as well as the probability that a dislocation reaction will terminate operation of these largest sources. It naturally follows that the resultant flow curves will vary randomly with these local rearrangements. At a fixed density, the stochastic response becomes more pronounced as the simulation cell size decreases, as there are potentially fewer sources that can operate at nearly the same stress, thereby maximizing the influence of any individual source. Also, the magnitude of the size-affected response is directly dependent on the initial density, as higher starting densities result in a weaker scaling response (more available sources) and vice-versa.[29] This dependence results in behavior that is counter to classical strengthening ideas, i.e., an increase in the initial density can *soften* microcrystals,[29] which has been confirmed experimentally.[38]

Conclusions

The micro-compression experiments and simulations described above have made a significant impact on the study of dislocation-based deformation processes. These studies have clearly demonstrated that size-scale effects exist independently of other previously known size effects such as nucleation-controlled deformation or the presence of imposed strain gradients. Notably, the close coupling of experiments and simulations have led to the discovery of new strengthening mechanisms that are active in micro-scale samples. The small sample volumes also facilitate the measurement of stochastic

dislocation activity (strain bursts), which has greatly aided the understanding of how dislocation ensembles dissipate energy, and hopefully will help advance new meso-scale deformation theories.

With continued effort, we predict that this type of experimental methodology will allow one to locally determine the full suite of properties of various microconstituents (phases, precipitates) in fully-processed engineering materials. Challenges in the area of sample fabrication and testing include developing parallelized fabrication methods that can be applied to a wide range of materials, and in performing a broader suite of testing modes (tension[39] and elevated temperature testing, for example). Similar areas of improvement exist for the modeling and simulation tools, such as incorporating realistic starting dislocation networks or internal microstructures, as well as simulating 'larger' volumes that truly bridge the gap between micro- and mesoscopic deformation.

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Figure Captions

Figure 1. (a) Schematic of the microcompression test (b) schematic of the flow response of a microcrystal oriented for single slip (c) scanning electron image of a 5 micrometer diameter microcrystal sample of pure Ni oriented for single slip (d) SEM image of (c) after testing. Figure is from Ref 18, and was originally adapted from Ref. 9.

Figure 2. Shear stress-shear strain plots of <269>-oriented pure Ni microcrystals grouped by sample diameter: (a) 18 micrometer and larger, (b) 10-11 micrometer, (c) 4-6 micrometer, and (d) 2.5 micrometer and smaller. The thick black curve in each plot is from a macroscopic crystal of the same orientation, and plots are truncated at 15 % strain for clarity. Figure is adapted from Ref. 9.

Figure 3. Composite plot of published microcrystal flow stress data as a function of sample diameter for FCC metals. The scaling exponent *n* that best fits the data is approximately 0.6. Figure is adapted from Ref 18.

Figure 4. Composite plot of published flow stress data for 3D DDS as a function of simulation cell size. Data in the plot have been normalized by the shear modulus and Burger's vector used for each study

(see Eq. 1). The light blue area designates the range of experimental measurements shown in Fig. 3. Figure is adapted from Ref. 18.

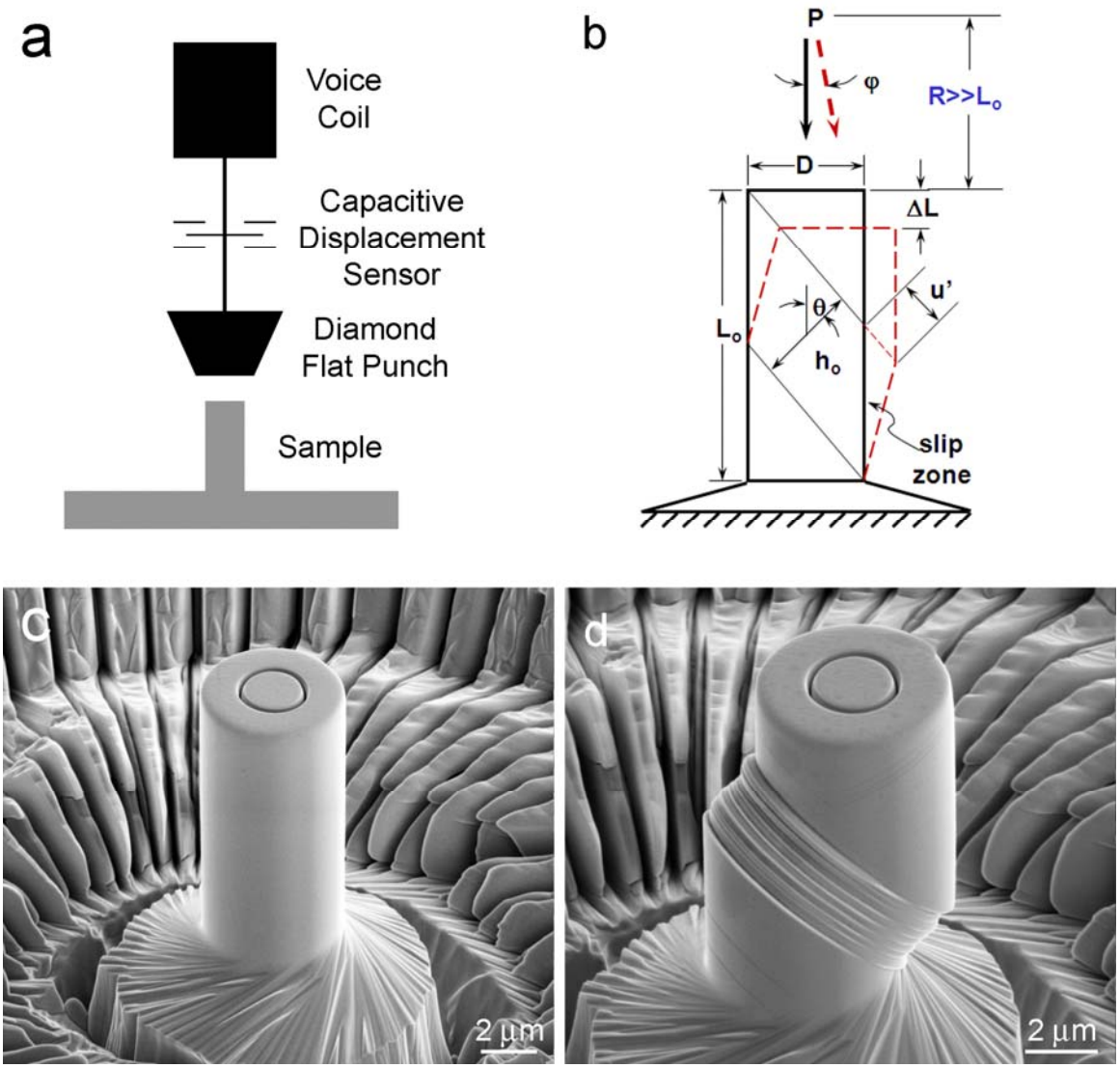


Figure 1

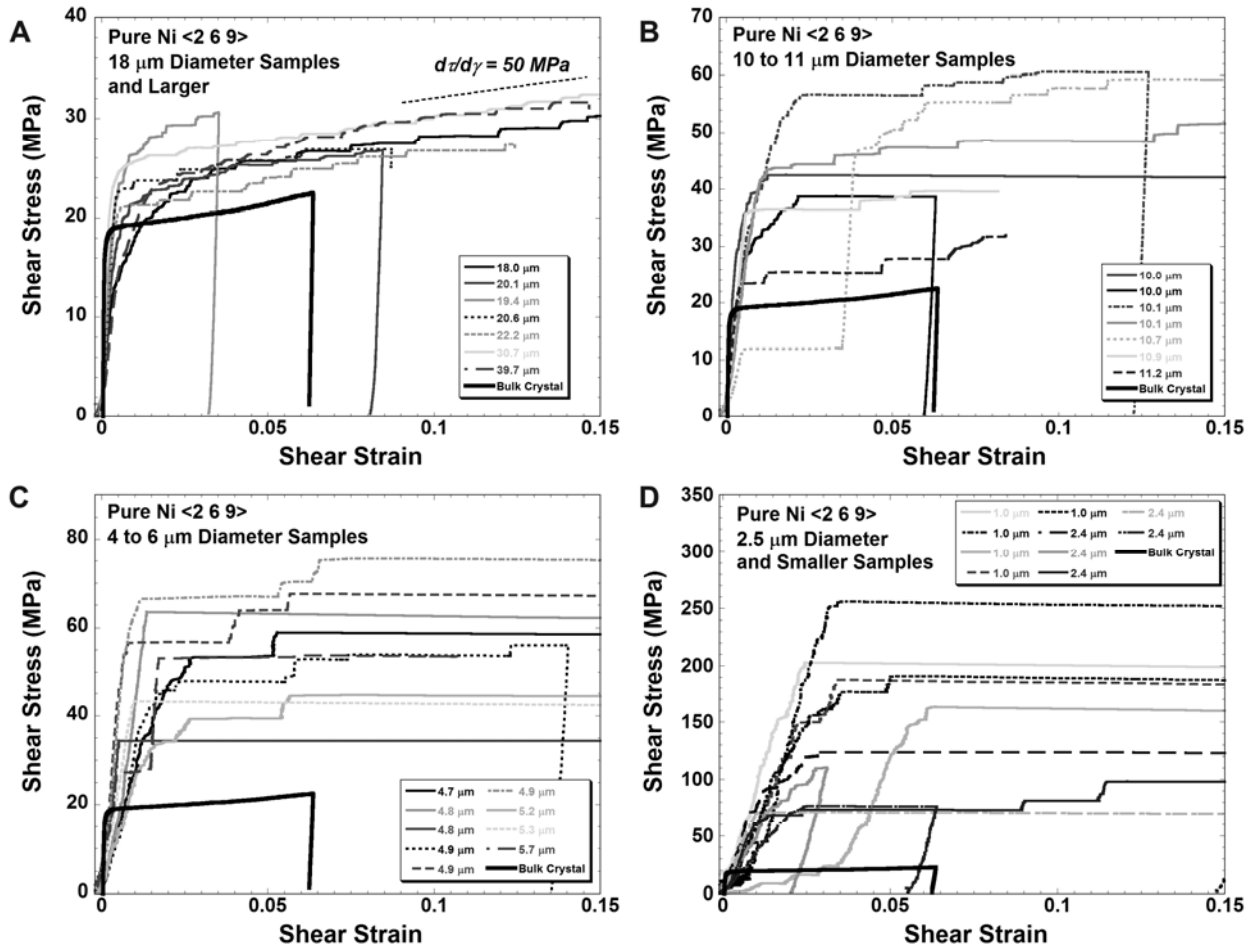


Figure 2

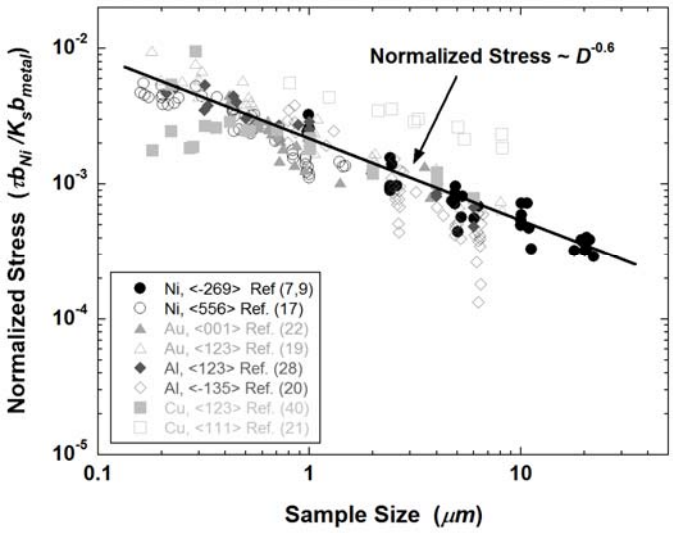


Figure 3.

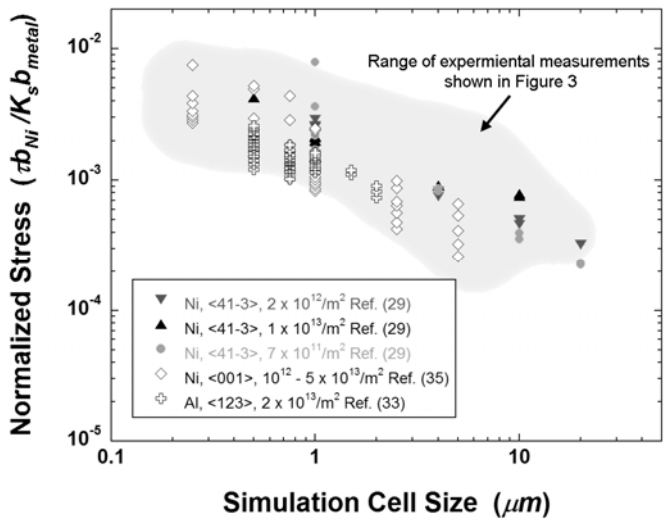
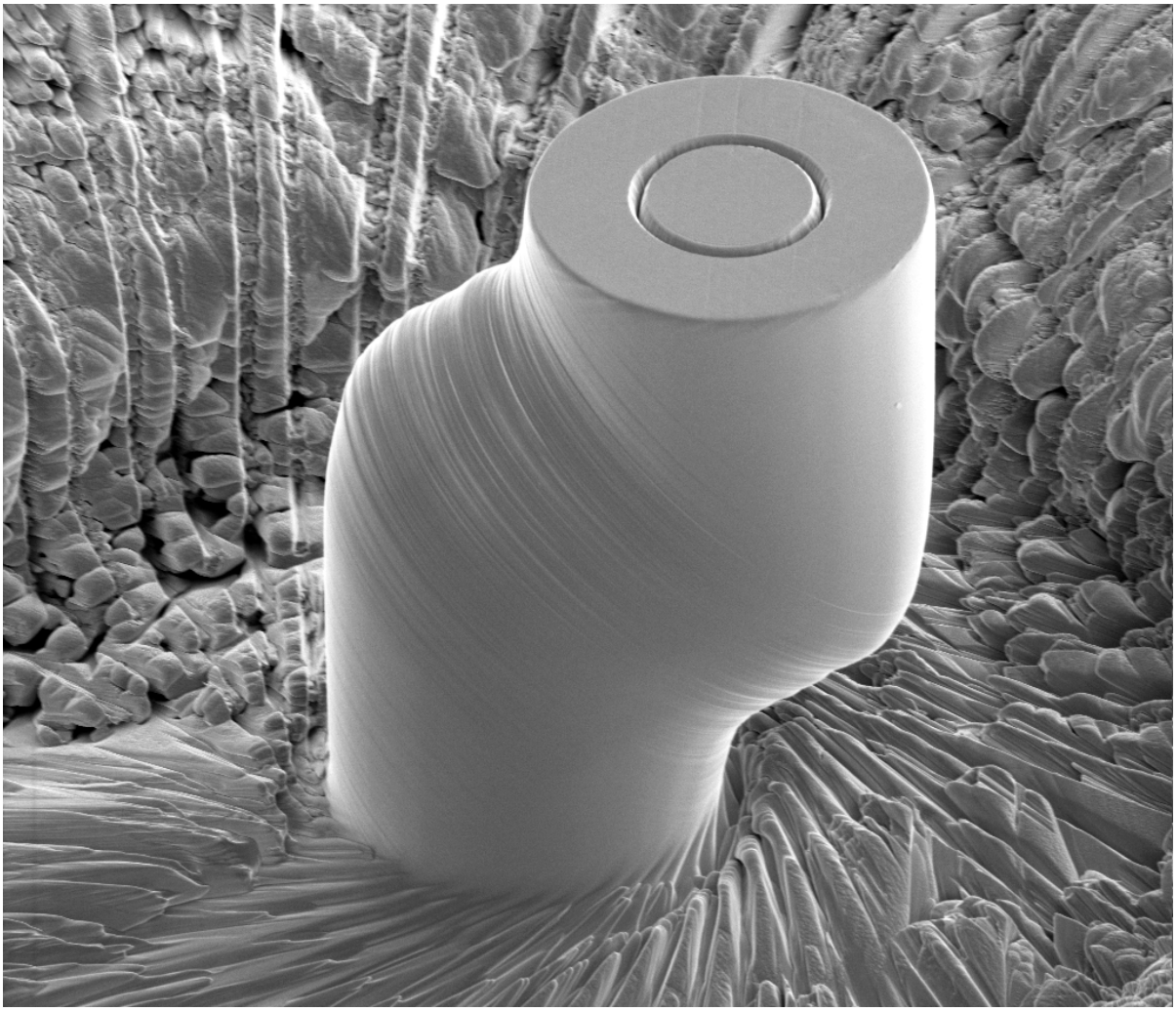

Range of expermiental measurements shown in Figure 3
Ni, <41-3>, 2 x 10^12/m^2 Ref. (29)
Ni, <41-3>, 1 x 10^13/m^2 Ref. (29)
Ni, <41-3>, 7 x 10^11/m^2 Ref. (29)
Ni, <001>, 10^12 - 5 x 10^13/m^2 Ref. (35)
Al, <123>, 2 x 10^13/m^2 Ref. (33)
Normalized Stress (πb_Ni /K_s b_metal)
Simulation Cell Size (μm)
10^-2
10^-3
10^-4
10^-5
0.1
1
10

Figure 4.



Cover Photo